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GENERATION OF CONTROLLED VAPOR CONCENTRATIONS OF CHEMICAL
AGENTS USING THE PORTABLE GENERATOR

by

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July 1978

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) (U) The portable generator is used to generate controlled vapor concentrations of chemical agents. Two factors that were seen in the use of the portable generator are: first, it is a process that is faster in equilibrating, and second it is a safer process because of the use of less or of more dilute agent. The portable generator is also much smaller than any previously used apparatus.		

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PREFACE

The work described in this report was authorized under Task 1M764725DO2201. The work was started in September 1972 and completed in July 1976. The data are collected in notebooks 8812, 9079, 9120, 9178, 9197, 9205, 9308, 9311, 9360, and 9393.

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CONTENTS

	<u>Page</u>
I. INTRODUCTION	7
II. EXPERIMENTATION	7
A. Portable Generator	7
B. Agents Generated	12
III. DISCUSSION	12
IV. CONCLUSION	15
DISTRIBUTION LIST	17

LIST OF FIGURES

Figure

1	Portable Generator	8
2	Flow Chart for Modified Portable Generator for Room Temperature Operation	9
3	Flow Chart for Modified Portable Generator for High Relative Humidity Work	10
4	Drawing of Standard Edgewood-Type Glass Bubbler	11

LIST OF TABLES

Table

1	Modified Portable Generator Parts List	13
2	Typical Room Temperature Generator Settings for Various Agents	16

GENERATION OF CONTROLLED VAPOR CONCENTRATIONS OF CHEMICAL AGENTS USING THE PORTABLE GENERATOR

I. INTRODUCTION.

In early development, while operationally testing agent alarms in the cold temperature chamber, the scientists were required to test the alarms with chemical agents. During their investigation, the scientists were to find a method of testing with agent that would be safe as well as easy to use. The chosen method was one which utilized a U-tube filled with agent laden alundum.¹ Later, Baldauf and Ong worked on a simplified version of the Q-5 agent generator, developing a device which they called a portable generator.² The portable generator, which was safer and stabilized more rapidly than a Q-5, was used to generate low concentrations of GB and VX for detector purposes.³ This portable generator was later modified for use in testing the XM256 detector kits.* Using this modified portable generator, various agents were generated to complete the XM256 detector kit testing.

II. EXPERIMENTATION.

A. Portable Generator.

The portable generator consists of four main parts: an air pump, valved flowmeters, a bubbler for agent or external agent source, and an agent chamber (figure 1).⁴

1. Air Pump.

The air pump, a small Gast-type vacuum pump, provides the desired volume of air that is necessary for the device to be tested. The air from this pump is divided into two parts: one part, which is dried by passing it through a drying column, is flowed through an agent-filled bubbler; and the second part, which is of greater volume, is used to dilute the dry agent-filled air (figure 2). Later in the XM256 detector kit testing, it was necessary to divide the air from the pump into three parts so as to obtain a high humidity environment (80% to 95% RH) (figure 3).

2. Valved Flowmeters.

The flowmeters are panel-mounted, high-accuracy needle-valve-type with both glass ball and stainless-steel ball floats. The flowmeter used to control the dry agent-carrying air indicates from 4 to 262 cubic centimeters per minute, and the flowmeter used for the dilution air indicates from 1.5 to 49.3 liters per minute.

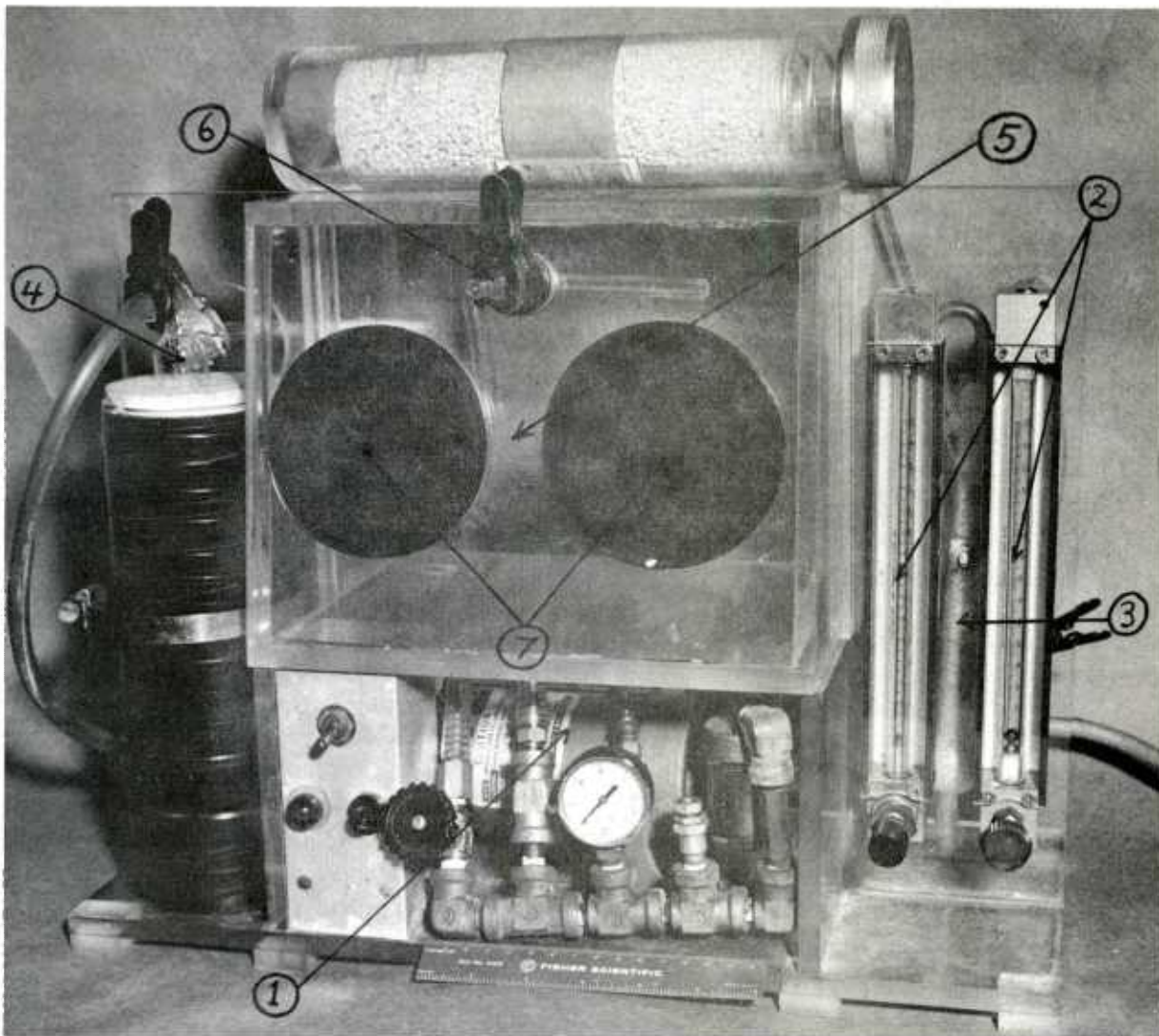
¹Manual 136-300-52E, 3 May 1976, Instruction Manual for the Installation, Operation, and Maintenance of Dilution Apparatus, Q5.

²Baldauf, F., and Ong, K. Patent Document No. EA 6501.

³Silvestri, A. EATM 2300-7. Generation and Detection of Low Concentrations of VX. August 1973.

⁴Manual 136-300-297, 10 November 1975, Instruction Manual for the Installation, Operation, and Maintenance of the Portable Gas Generator, 0219.

*Packard, M., and McDowell, C. Personal Communication.



KEY

1. PUMP
2. FLOWMETERS
3. DRYING COLUMN
4. AGENT BUBBLER
5. AGENT CHAMBER
6. SAMPLING TUBE (FOR CONCENTRATIONS)
7. SAMPLING PORTS (FOR INTRODUCTION OF XM256 KITS)

Figure 1. Portable Generator

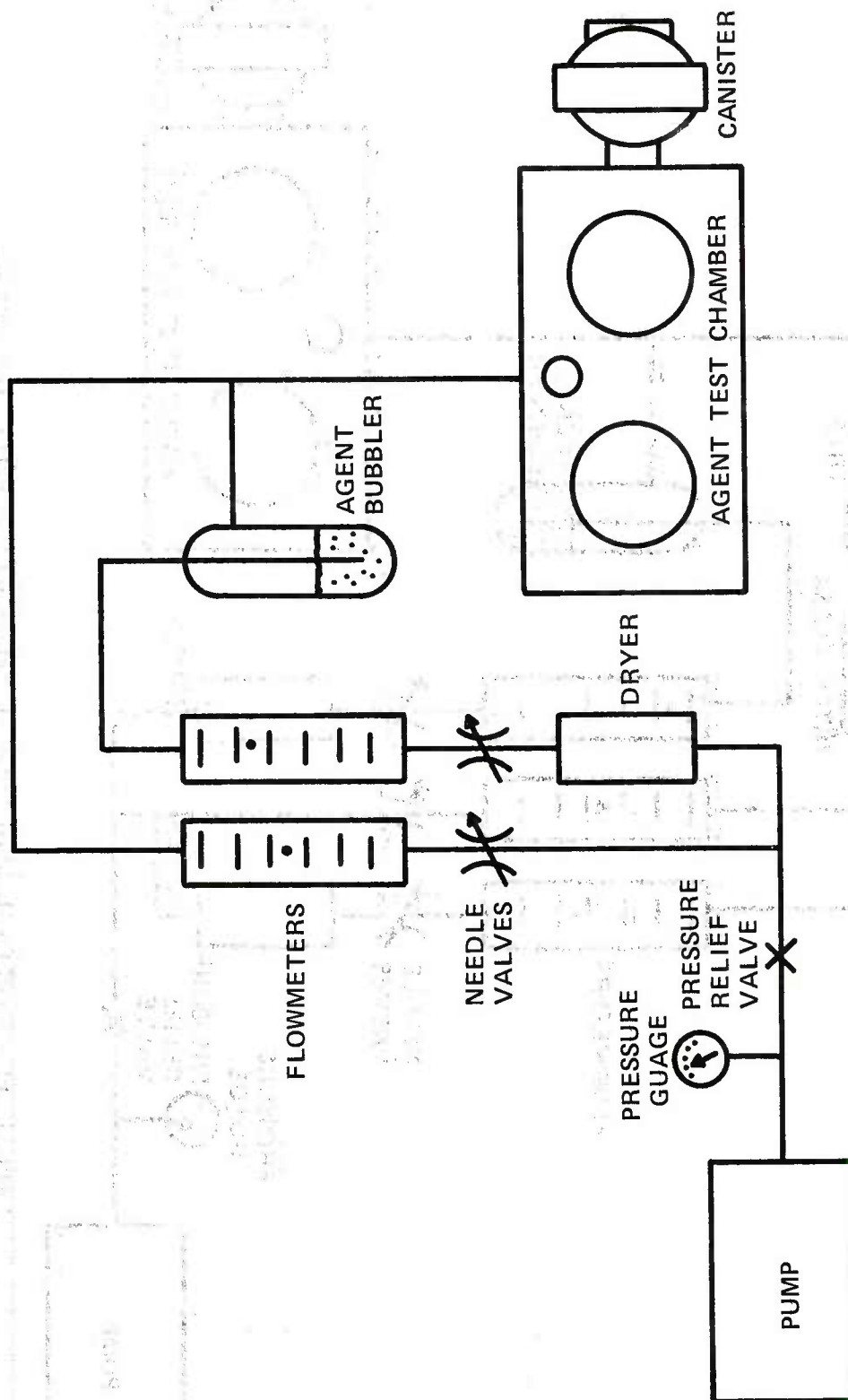


Figure 2. Flow Chart for Modified Portable Generator for Room Temperature Operation

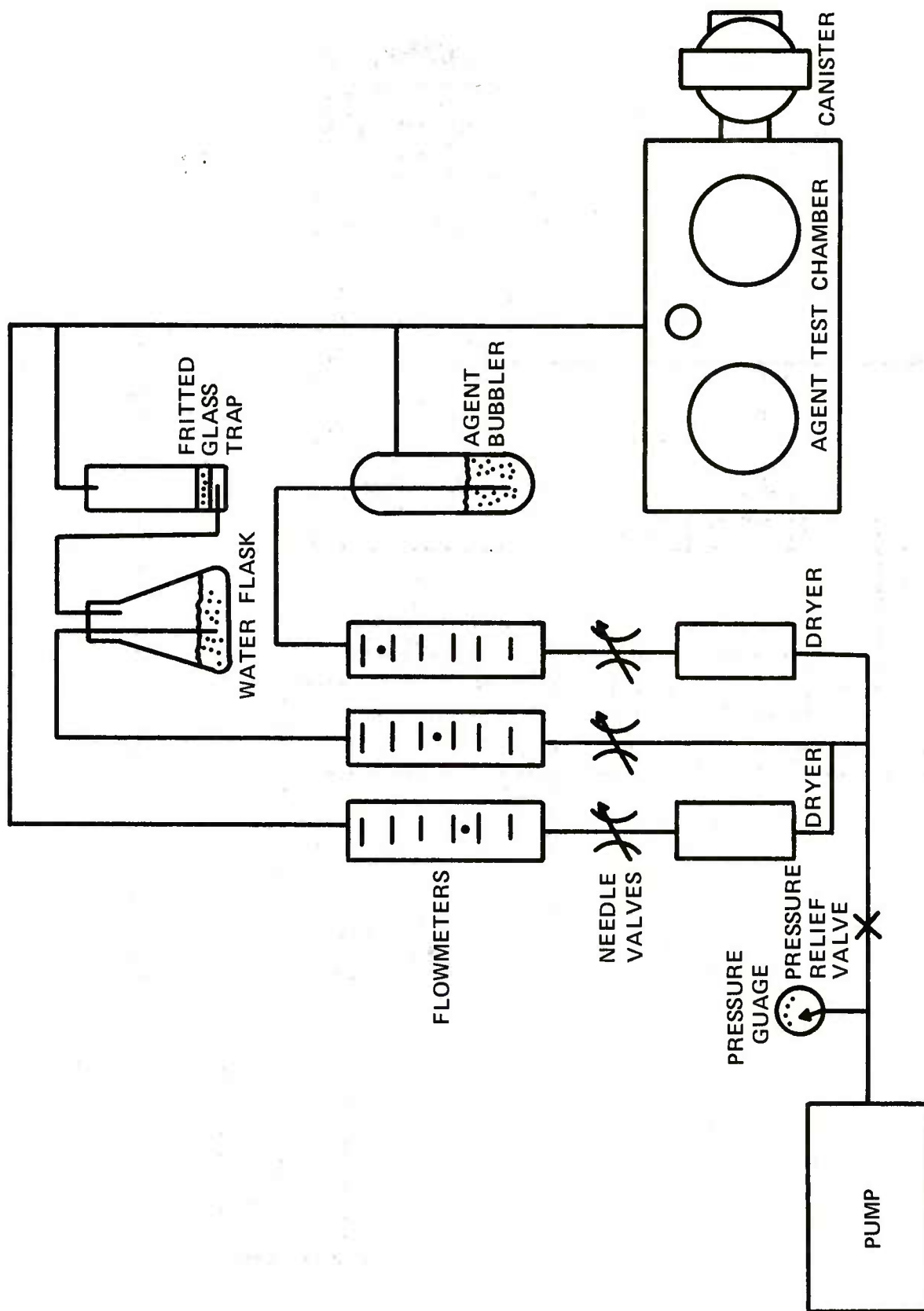


Figure 3. Flow Chart for Modified Portable Generator for High Relative Humidity Work

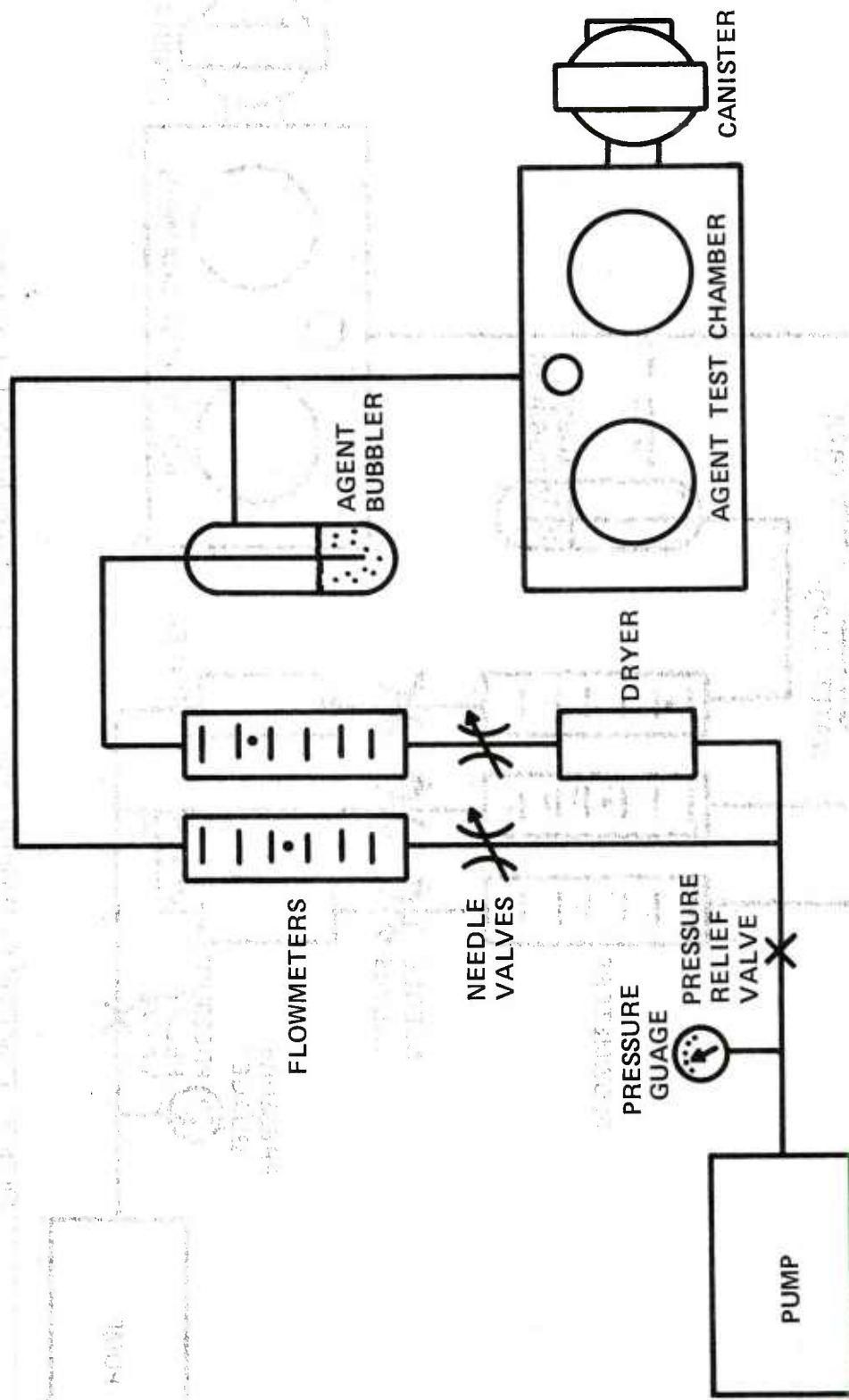


Figure 2. Flow Chart for Modified Portable Generator for Room Temperature Operation

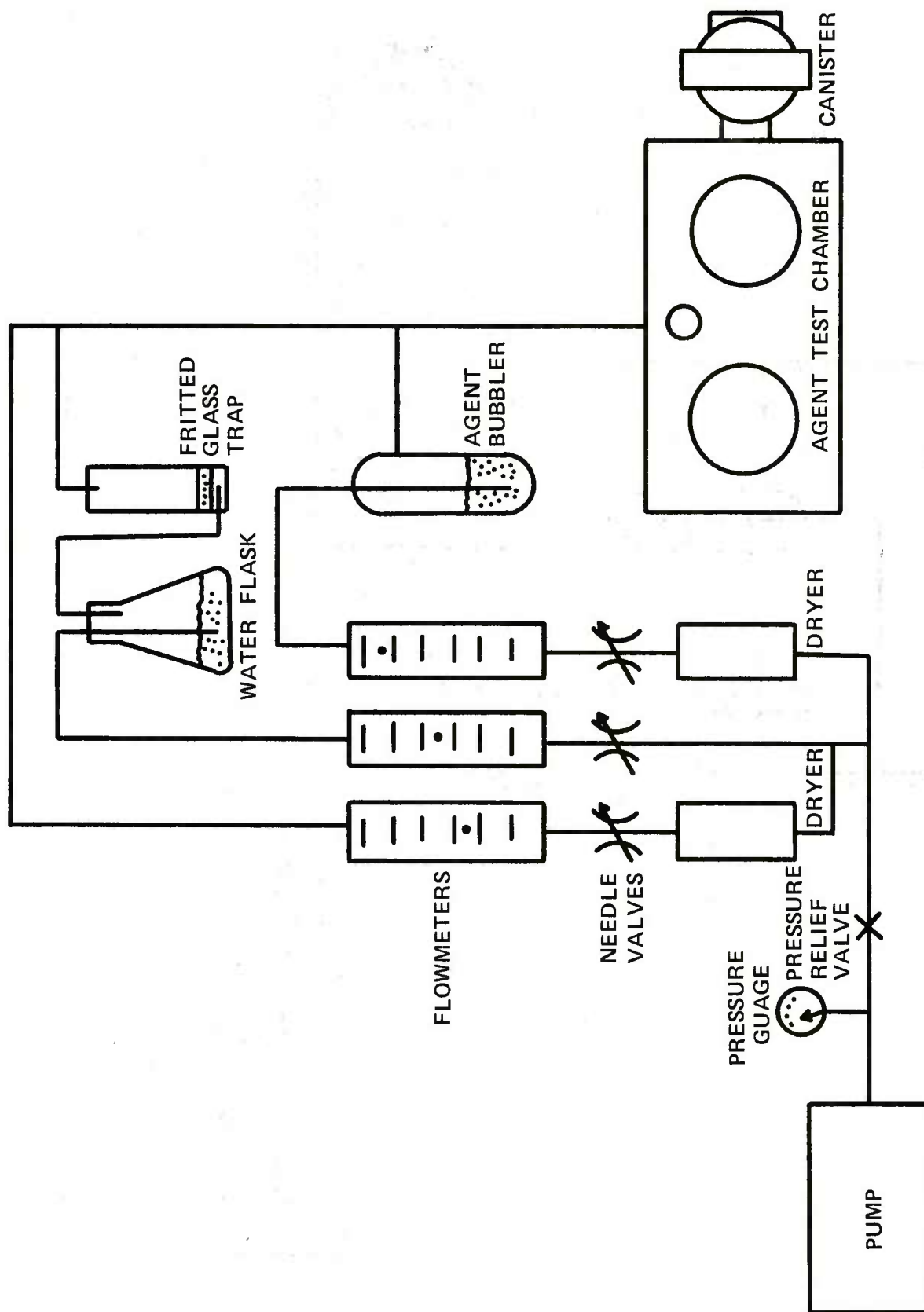


Figure 3. Flow Chart for Modified Portable Generator for High Relative Humidity Work

3. Bubbler for Agent or External Agent Source.

The bubbler used for the agent supply is a standard Edgewood-type, approximately 5 inches long and 1 inch in diameter (figure 4). The air enters the top of the bubbler, bubbles through the agent, and discharges through a side arm of the bubbler into the chamber.

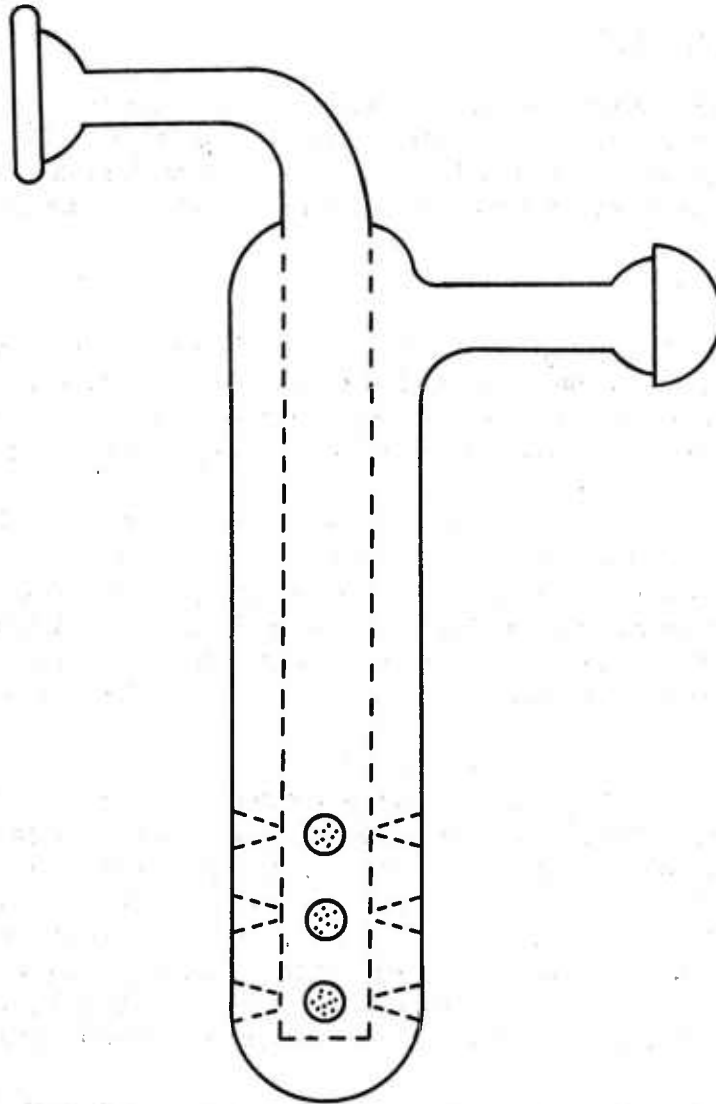


Figure 4. Drawing of Standard Edgewood-Type Glass Bubbler

4. Agent Chamber.

The agent chamber (figure 4) part of the generator is a box of clear acrylic sheet approximately 10 inches long, 8 inches high, and 7.5 inches deep. The chamber is connected to the agent supply with a glass socket joint (12/5) to which a glass T is connected; the agent-carrying air and the dilution air flow to the two branches of this tee. An M11 combat canister is connected to the discharge part of the chamber, through which the excess agent-diluted air flows. The system is

noncontaminating since all agent is removed from the effluent by means of the canister. The front of the chamber has a sloped face with three holes, two of which are large and are fitted with No. 15 rubber stoppers; the third hole, cut in the top center portion of the front, is fitted with a small stopper (No. 5) which has a glass angle tube with a glass ball joint (12/5) on the end for external agent sampling. The large holes are used for insertion and withdrawal of detector devices. A complete list of parts for a portable generator is given in table 1.

B. Agents Generated.

The various agents used in the portable generator were determined by the requirements for testing of the XM256 detector kit. These agents were as follows: GA, GB, GD, VX, AC, CK, CX, HD, and L. The above agents had such a varied vapor pressure that each had to be handled differently in order to generate the required concentrations in the portable generator.

III. DISCUSSION.

The portable generator was first developed for testing various agent alarms; later it was modified for use in testing the XM256 detector kit. The most notable improvements that the modified portable generator have over previously used equipment include: (1) time to reach a stable concentration, (2) size and compactness, and (3) safety.

In past years, most agents were generated at Edgewood Arsenal (now Chemical Systems Laboratory) with a Q-5 dilution apparatus. Many times this type of generator had to be charged with agent at startup and allowed to run for a period of time (from a few hours to a few days) until the agent concentration was stabilized. Often, if a change in concentrations were desired, it would again mean a long wait until the new concentration was stabilized. Using the portable generator, one can start the generator in the morning of a test and have a stabilized concentration in 1 hour or less.

In comparing the older Q-5 generator and glove box with the modified portable generator, a great difference is seen. The Q-5 generator is 16 inches long, 13 inches wide, and 28 inches high. The Q-5 generator was then connected to a glove box whose dimensions were 25 inches long, 12 inches wide, and 24 inches high. Together, these two pieces occupied a large volume. In comparison, the modified portable generator is 18 inches long, 8 inches wide, and 14 inches high. In almost every dimension, the modified portable generator is smaller than either of the two combined pieces of previously used equipment. This smaller size was especially useful later, when testing of the XM256 detector kit had to be done in an environmental chamber.

Safety in using the portable generator is also a consideration. In most cases, the Q-5 generator used an agent holder which required approximately 20 to 25 milliliters of agent. The portable generator was designed to use agent in a bubbler to generate concentrations and, in most cases, the agent bubbler contains a dilute mixture of agent and a carrier (solvent). When neat agent was required, a mini-bubbler was used in order to minimize the amount of agent actually used, usually 1 to 2 milliliters. When using the portable generator, it should always be used in a laboratory hood with a hood face velocity of at least 150 ± 30 feet per minute. Also of note is the exit canister of the generator; a filter-use record should be kept, and the filters should be changed at regular intervals (not more than 1 year, depending on frequency of use).

Table 1. Modified Portable Generator Parts List

1. Air Compressor, motor mounted, oil-less type, 1/15 horsepower, 3450 RPM, Gast Manufacturing Corporation Model No. 0531-102-347 (1 each).
2. Flowmeter, panel mount, w/standard needle valve, stainless steel, 1/4-inch tubing connection. Equipped with a No. 604 tube including a pyrex and stainless steel float, 0.4 to 16.9 liters of air per minute, with an NRS orifice No. 5. Matheson Cat. No. 621 PSV (1 each).
3. Flowmeter, panel mount, w/NRS high accuracy needle valve, stainless steel, 1/4-inch tubing connection. Equipped with a No. 601 tube including a pyrex and stainless steel float, 4 to 262 cc of air per minute, with NRS orifice No. 2. Matheson Cat. No. 620 PSX (1 each).
4. Pressure Relief Valve, Dees Hydraulics Company, No. AA 203 (1 each).
5. Pressure Gauge, Dees Hydraulics Company, No. AA 642 (1 each) Street.
6. Cajon Elbow (SE), 1/8-inch female pipe size, Part No. Z-SE (3 each).
7. Cajon Street Tee (ST), 1/8-inch pipe size, Part No. Z-ST (2 each).
8. Cajon Tee (T), 1/8-inch female pipe size, Part No. Z-T (1 each).
9. Cajon Hex Long Nipple (HLN), 1/8-inch male pipe size, Hose inside diameter, 1/4-inch, Part No. Z-MHC-4 (2 each).
10. Cajon Male Hose Connector (MHC), 1/8-inch male pipe size, Hose inside diameter, 1/4-inch, Part No. Z-MHC-4 (2 each).
11. Drying Tube, Calcium Chloride, Polypropylene, 8 inches long, Will Scientific Cat. No. 12155 (1 each).
12. Tygon Tubing, 1/4-inch inside diameter, 3/8-inch outside diameter (3 feet).
13. Chassis, Electrical, Aluminum, "Mini-Box", 5-inch \times 2-1/4-inch \times 2-1/4-inch. Allied Cat. No. 736-3655 (1 each).
14. Cable, Power, Rubber Jacketed, Size 3-18, Allied Cat. No. 732-2113 (6 feet).
15. Plug, Power Cord, 3 wire, Allied Cat. No. 785-0203 (1 each).
16. Holder, Fuse, for 1/4 \times 1-1/4 fuse, Allied Cat. No. 845-0092 (1 each).
17. Fuse, Slow Blow, A/3AG/MOL, 3 AMP, Allied Cat. No. 845-0235 (1 each).
18. Pilot Light, Neon, Clear Lens for 1/2-inch Hole, Solder Tabs, Allied Cat. No. 844-5135 (1 each).
19. Switch, Off-On, SP-ST with Solder Terminals, 15/32-inch Diameter Bushing, Allied Cat. No. 757-7040 (1 each).
20. Tubing, Heat-Shrinkable, Polyolefin, 1/4-inch Diameter, Allied Cat. No. 708-3943 (6-inches).

Table 1. (Continued)

21. Grommets, Rubber, 1/4-inch-diameter hole for mounting in 3/8-inch-diameter hole (2 each).
22. Flask, Vacuum, DEWAR, Capacity 665-ml, overall height 9-1/4-inch; 2-3/4-inch inside diameter. Fitter w/insulated (Polystyrene) Stopper 1-1/2-inch-thick containing center-bored hole 1-inch-diameter, Prieser Scientific Company Cat. No. 12-7510/02 (1 each).
23. Rubber Stoppers, Size No. 15, Will Cat. No. 23390 (2 each).
24. Rubber Stoppers, One-Hole, Size No. 5, Will Cat. No. 23391 (1 each).
25. Hose Clamp (1 each).
26. Clamp to hold drying column (1 each).
27. Ground Glass Ball Joint, 12/5 size, 6 inches long bent at 2-1/2 inches from ball to approximately 45° (1 each).
28. Ground Glass Socket Joint, 12/5 Size, 2-1/4 inches long (1 each).
29. T Joint with Ground Glass Sockets at Side and Bottom of T and Ball at other side of T, Top of T 2-1/2 inches long and Leg of T 3 inches long (all joints 12/5 size) (1 each).
30. L Ground Glass Sockets, 12/5 Size, Joints on one end only and legs are 1-1/4 inches long (2 each).
31. Clear Acrylic Sheet Agent Chamber Box (1/4-inch Acrylic) 10 inches long, 8 inches high, 7 inches deep at top and 8 inches deep at bottom (1 each).
32. Clear Acrylic Sheet Frame for Holding Agent Chamber, Pump, Flowmeters, and other miscellaneous parts (1/4-inch Acrylic) 18 inches long, 10 inches deep, 13-3/8 inches high (without feet) (1 each).
33. Critical Orifice (Brass Tube 1-1/2 inches long and 5/16 inches in diameter Blanked Off at One End) Hole in one end to allow no more than .3 liter of air to pass through (1 each).

OPTIONAL PARTS FOR HUMIDIFIED GENERATOR

1. Flowmeter, Same as No. 3 above (1 each).
2. Cajon Street Tee (ST), 1/8-inch Pipe Size, Part No. 2-ST (1 each).
3. Cajon Street Elbow (SE), 1/8-inch Female Pipe Size, Part No. 2-SE (1 each).
4. Nupro Valve, 1/8-inch Male Pipe, Part No. B-2J2 (1 each).
5. Cajon Female Hose Connector (FHC), 1/8-inch Female Pipe Size, Hose inside diameter 1/4-inch, Cajon Part No. 2-FHC-4 (1 each).
6. Drierite, Gas Drying Unit, Will Scientific Inc. Cat. No. 12148 (1 each).
7. Filtering Flask, 2000-ml Size (1 each).
8. Tygon Tubing, 1/4-inch inside diameter, 3/8-inch outside diameter (1 each).
9. Fritted Glass Water Trap (1 each).

When generating various agents in the portable generator, a variety of techniques and settings were used. For all nerve agents, with the exception of VX, a very dilute mixture of from 1% to 5% of agent and either hexylene glycol or propylene glycol was used. The blood gas AC was first generated from a 20% aqueous KCN solution, and later from a HCN-air mixture cylinder (1500 ppm). When generating the blood gas CK, the neat agent was used. Generation of the blister agents HD and CX was done again using dilute solutions of agent. For HD, the solution was 20% HD in hexylene glycol and for CX, the solution was approximately 20% CX in water. The only neat agents used in the generator were the nerve agent VX and the blister agent L. Some of the typical portable generator settings for a given concentration at room temperature are listed in table 2.

IV. CONCLUSION.

The portable generator can be used to generate vapors of chemical agents at controlled concentrations. The use of this type of generator allows a stable concentration to be generated in a shorter time than the older Q-5 generator. The safety factor is another important advantage in the use of the portable generator since a smaller amount of agent is used either neat or in a diluted solution. Also, the generator is much smaller than apparatus previously used. Because of these factors, the portable generator should be used to produce any controlled vapor concentrations of almost any chemical agent.

Table 2. Typical Room Temperature Generator Settings for Various Agents

Agent	Agent air	Dilution air	Concentration
	cc/min	ℓ/min	mg/m ³
GA	110	3.0	0.9
GB	15	13.5	0.04
GD	20	5.0	0.1
VX	40	3.9	0.08
AC	45	6.4	10.0
CK	7	7.0	100-200
HD	24	5.6	3.0
CX	13	7.2	3.8
L	35	3.0	13.0

NOTES: GA - 5% in hexylene glycol use 85°F water bath.

GB - 2% in propylene glycol use ice bath.

GD - 5% in hexylene glycol use 85°F water bath.

VX - neat agent use 110°F water bath.

AC - gas cylinder mixture (about 1500 ppm).

CK - neat agent use ice bath.

HD - 20% in hexylene glycol use 85°F water bath.

CX - 5% in water use ice bath.

L - neat agent use ice bath.

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